

# PATENT SPECIFICATION

418,349

Convention Date (Norway): April 3, 1933.

Application Date (in United Kingdom): March 26, 1934. No. 9450/34.

Complete Accepted: Oct. 23, 1934.

COMPLETE SPECIFICATION.



## Improved Method of Maintaining a High and Dense Suspension of Crystals and like Granular Solids in a Liquid.

We, AKTIESELSKAPET KRYSTAL, a Norwegian Company, of Kronprinsens gate 17, Oslo, Norway, do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

This invention relates to processes of the type in which a suspension of granular solid substances is subjected to the action of a flowing liquid, and the invention has for its object a method of conducting the treatment of such suspensions by means of which it is made possible to retain the substantial portion of the granular matter in a confined suspension space, and in effective contact with the flowing liquid during the treatment.

A known method of retaining an actually dense (static) suspension of granules in an ascending flow of liquid in a crystallizer, a dissolving apparatus or a lixiviating apparatus is that disclosed in applicant's Norwegian patent specification No. 40,114 (corresponding to British patent specification No. 219,301).

By means of the method which is the subject of the present invention substantially the same results as according to the said known process are achieved in a more simple manner.

In known apparatus for carrying out a process in which a suspension of granular solid substances is subjected to the action of a flowing liquid, a vessel has a vertical annular partition with a propellor stirrer to circulate liquor down the central space and up the annular space. To the bottom of the vessel an elutriating tube is attached down which the coarser crystals descend. The central space of the vessel, moreover, has a large cross-sectional area i.e. a large fraction of the total cross-sectional area of the vessel.

The characteristic feature of the present invention consists therein that a flow of liquid is to its greater part introduced into the suspension to be treated just above a substantially impervious bottom in a suspension chamber as a stream or streams, having a total and maximum sectional area which is only one third of the horizontal sectional area of the suspension

chamber, the said streams being introduced with sufficiently high velocities to bring about a coherent overturning movement of the entire body of granules in a direction which along the bottom moves from the entrance openings.

An embodiment of the invention is described in the following description, reference being made to the accompanying drawing, in which:

Figure 1 is a diagrammatical sectional view of an apparatus adapted for carrying the invention into effect.

Figures 2—8 are diagrammatical views of modified arrangements.

In the example illustrated in Figure 1 a circulating flow of liquid is maintained in a container 2 by means of a propeller pump 3 which forces the liquid down a tube 4 and up through a suspension of granules 5, as indicated by arrows. The container 2 is provided with a cooling jacket 1.

In the operation of this apparatus a large coherent overturning movement of the entire body of granules is brought about in spite of the fact that the layer of granules is high and dense. The reduction in pressure and in consequence thereof of the horizontal propelling power produced by the liquid on its being forced out through the dense collection of granules in front of the entrance opening below tube 4 is here in fact much larger than in the case of the same quantity of liquid having entered as an unobstructed flow and only afterwards being caused to impinge upon a body of granules as is the case in several prior processes. Because, if one conceives of a high heavy column of granules, sinking downwards along the tube 4 as indicated in arrows, it is obvious that the liquid will have to be forced out through the sectional area between the granules which is only a small fraction of the whole of the free sectional area below the tube 4, the hereby produced horizontally directed super pressure in the opening exerting even in some distance outside of the same a more strongly accelerating influence on the body of the granules in a horizontal direction where it is readily movable than in a vertical direc-

tion, where the column of granules not only by its weight, but also by its being deflected from a descending to a horizontal movement, will take up the upward static pressure component of the liquid. The consequence of this is that the outwardly directed flow of liquid is followed at the bottom by a still more rapid flow of liquid. At the wall 2 therefore more liquid will be flowing than along the tube 4, so that the granules will there be floating farther apart and the weight per litre of suspension will be less at 2 than at 4. This difference in weight per litre at the centre and at the periphery will effect a great increase in the power producing the overturning motion. In addition hereto comes the important fact that the liquid in the course of the flow of granules along the bottom also will ascend and hereby will maintain the high and heavy body of granules floating in its entirety so that it is not capable of exerting any considerable friction producing pressure in its interior and against the bottom. An overturning movement of the entire body is thereby made possible. This has not been the case in other known types of apparatus, except in the arrangement disclosed in Norwegian patent specification No. 40,114 (corresponding to British patent specification No. 219,301).

It is known to bring about an overturning movement of a quite low and loose suspension by means of a unilateral entrance flow of liquid and a vaneshaped projection on the opposite wall, which deflects the ascending flow at the wall and converts it to a horizontal flow at the top of the suspension. In the case of a high dense suspension as that employed according to the present invention, such a vane would be unnecessary and harmful. In the parts of the suspension which are situated near below the vane, the vertical velocities of the liquid maintaining the granules in suspension are made impossible. The heavy tight body of granules as a consequence thereof would sink and thereby destroy the above mentioned powers which should function to move the ascending flow at this point, and which are upheld according to the present invention by permitting the flow of liquid to pass further along upwards above the suspension.

To this is to be added that the substance to be suspended in actual practice always will consist of granules of different sizes, and that a vane on the wall of the container will narrow the upper sectional area of the suspension where even the granules of the smallest size are prominently present, so that a large part of same will be drawn out of the suspen-

sion chamber.

As contrasted herewith, in accordance with the present invention, it is an important characteristic that the predominant part of the various sizes of granules present in the suspension is not drawn along out of the suspension chamber, because the flow of liquid is distributed over the entire sectional area of the container. Already at the bottom the flow is forced transversely over the entire cross sectional area of the container.

It is also known to cause a flow of liquid to enter through a single opening in the lower part of a container below a layer of granules. But in such a case the container has been constructed, not as according to the present invention with large sectional area already near the entrance opening, but intentionally funnel shaped, and only so small quantities of inflowing liquid have been employed that the flow of liquid and granules will ascend at the centre and descend along the wall of the funnelshaped container. Such a method involves great inconvenience, particularly in crystallizers. The liquid flows upwards only in a quite small sectional area at the centre, and practically nothing passes upwards in the predominant peripheral part of the body of granules, so that only a quite small fraction of the surface of all the granules will be rationally utilized. Any real suspension of the entire body of granules is not produced. In addition, owing to the fact that the descending flow of granules at the wall of the funnelshaped container will be quite compact and have a much larger sectional area than the ascending flow, a great number of granules may happen to be lying dead upon one another for such a time that in a crystallizer they will begin to grow together or cake in the supersaturated solution. This effect will be accentuated thereby that the movement in an oblique direction inwards is retarded by "vault"-formation of the granules, because the mass enters a space which constantly decreases in size towards the axis of the funnel.

In contradistinction to various known arrangements for the maintenance of a suspension, the essential feature of the present invention consists therein that the total transverse sectional area of the entrance opening for the flow is only a fraction of the horizontal sectional area of the suspension chamber.

In the arrangement illustrated in Figure 2, the suspension container is presumed to be constructed with comparatively larger diameters. The drawing shows only the lower part of container 2 and tube 4. Above the bottom is placed

a circular disc 6 with a hole 7 in the centre, so that the flow of liquid from the tube 4 passes in a substantially horizontal direction out into the suspension, partly above and partly below the disc. Instead of one single disc, several discs may be employed to distribute the entering flow more uniformly in containers with large diameters.

In the embodiment illustrated in Figure 3 the slot below the supply tube is given different heights at different points of the periphery. This has the effect of producing local secondary flows, which by certain sizes of granules and container diameters increase the security against the formation of "wells" in the suspension. Instead of a continuous corrugated slot it is also possible to employ a series of separate holes.

Figure 4 shows the lower part of a suspension vessel in which a flow of liquid is forced from below up through an opening 9 in the bottom 10 and deflected outwards by a hood 11. This arrangement is of particular advantage, when the process is made use of by crystallisation in connection with evaporation for example by vacuum cooling or where the solution is heated by means of a chemical reaction in the crystallisation vessel, because the arrangement provides for a free surface for evaporation.

In this arrangement it may be said that the suspension chamber has an impervious or tight bottom inasmuch that the space in the hood 11 does not contain any suspension, that is to say, it forms no actual part of the suspension chamber.

Instead of forcing a flow of liquid horizontally outwards along the bottom from a central tube, one or more jets may also be forced horizontally inwards along the bottom from an annular chamber around the circumference of the vessel.

As indicated in Figures 5 and 6 as well as 7 and 8 in vertical and horizontal sectional views also other sectional forms of the vessel than the circular form may be chosen, such as for example a rectangular form.

In the case of a circular container section, the entrance opening must be annular or consist of annular rows of holes, in order that the jets shall be distributed over the entire sectional area of the bottom. In analogy herewith the jets should in the case of rectangular sectional forms, be so distributed as to sweep over the whole sectional area. The safest way of avoiding formation of "wells" in the suspension is to introduce the liquid horizontally or in a somewhat slanting direction upwards. The angle with the horizontal should preferably not be over 45

degrees.

In addition to the above mentioned and illustrated main openings for the entrance of liquid along the bottom, one may of course also arrange secondary openings, for example small holes in the disc 6, in Figure 2, where special conditions make it desirable to produce vivid motion of the granules.

A common feature of the various arrangements according to the present invention consists therein that the bottom is substantially tight. When holes are at all arranged in the bottom, the total sectional area of such holes measured immediately at their opening into the large suspension space will only be a small fraction of the sectional area of the suspension chamber.

The horizontal sectional areas of the suspension space need not be the same all over. It will frequently be of advantage to make the sectional areas larger in the upper parts of the suspension than in the lower. If the difference in size of the suspended granules is very large, the smallest granules will then not so readily be drawn along out of the suspension chamber.

A very important application of the above described process is in crystallisers, because in connection with an intensive production on a commercial scale of large quantities of granules growing in a supersaturated liquid, the process allows of separate growth of the individual granules without causing the granules to grow together or cake or to enclose liquid and dirt owing to dead points in the suspension.

Another important application of the process described above is to dissolve or lixiviate a granular crude material for example crude potassium chloride.

In the case of the invention being used in crystallisers and the supersaturation of the solution is brought about by a chemical reaction as for example between  $\text{NH}_3$  and  $\text{H}_2\text{SO}_4$ , a strong flow of liquid into and out of the suspension chamber is frequently not needed. It may then sometimes be sufficient to bring about the floating and overturning of the crystal suspension by forcing a flow of gas, air or steam instead of a flow of liquid or together with a comparatively small flow of liquid in through the entrance openings.

Having now particularly described and ascertained the nature of our said invention and in what manner the same is to be performed, we declare that what we claim is:—

1. In the treatment of granular materials with liquids a method of main-

- taining a high and dense suspension of granules floating in a flowing liquid, which comprises the step of injecting a fluid into a suspension of granules enclosed in a confined suspension chamber having a substantially imperforate or tight bottom, the said fluid being injected into the chamber at a place immediately above the said tight bottom, the total and maximum cross sectional area of the current of injected fluid at the point of entrance being only one third of the horizontal sectional area of the suspension chamber, the fluid being injected with sufficiently high velocity to bring about a coherent overturning movement of the entire body of granules in a direction which follows the bottom of the suspension chamber in a direction away from the points of entrance of the injected fluid.
2. Process according to claim 1, in which the injected fluid is of the same character as that in which the granules are suspended.
3. Process according to claim 1, in which the injected fluid is a gaseous material.
4. Process according to claim 1, in which the suspension chamber has a circular cross sectional area, the injected fluid being introduced into the chamber in a substantially radial direction.
5. Process according to claim 1, in which the suspension chamber has a rectangular sectional area, the propelling fluid being injected into the chamber in a direction parallel to the side walls of said chamber.
6. Process according to claim 1, carried into effect in a suspension chamber, having a circular cross sectional area, the propelling fluid being introduced into the suspension as a flow having its cross sectional area distributed concentrically around the axis of the chamber and with a direction of flow at the bottom from the centre outwards.
7. Process according to claim 1, carried into effect in a suspension chamber with rectangular cross sectional area and in which the fluid is injected into the suspension through one of the side walls and distributed over practically the whole breadth of the wall.
8. Process according to claim 1, carried into effect in a suspension chamber of rectangular cross sectional area, and in which the fluid is pressed into and through a vertical channel with rectangular sectional area disposed in the centre of the chamber, the fluid being caused to be injected into the suspension chamber, from the said channel in all directions along the bottom of the chamber.
9. Process according to claim 1, in which the treated suspension is a collection of growing crystals suspended in a supersaturated solution.
10. Process according to claim 1, in which the injected fluid is in part a liquid and in part a gaseous material.
11. Apparatus for treating a suspension of granules with a liquid, comprising a suspension chamber with a substantially tight bottom, means of injecting a fluid into the suspension chamber immediately above the said bottom through entrance openings, having a total cross sectional area which is only a small fraction of the horizontal sectional area of the suspension chamber.
12. Apparatus for treating suspensions of granules of soluble substances in liquids substantially as hereinbefore shown and described.
- Dated this 26th day of March, 1934.  
 AKTIESELSKAPET KRYSTAL,  
 Per Boulton, Wade & Tennant,  
 111/112, Hatton Garden, London, E.C. 1,  
 Chartered Patent Agents.

Fig. 1.

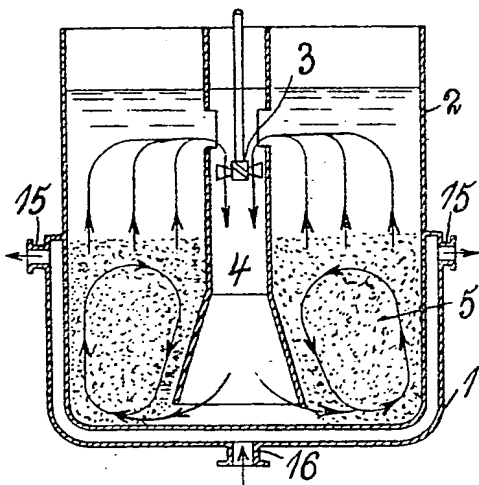


Fig. 2.

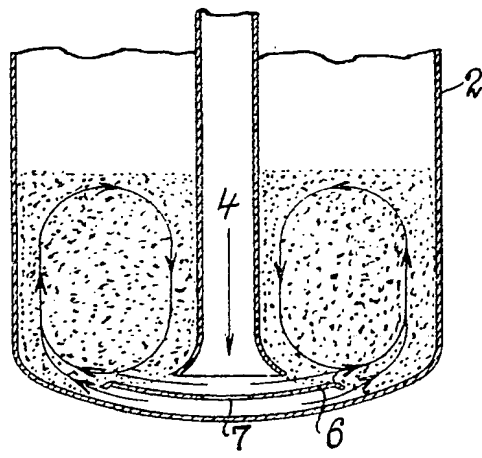


Fig. 3.

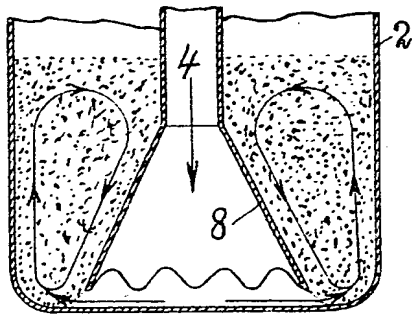


Fig. 5.

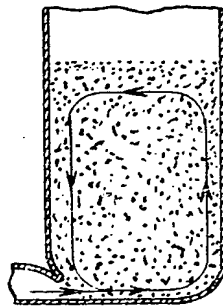


Fig. 7.

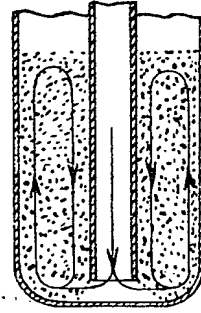


Fig. 4.

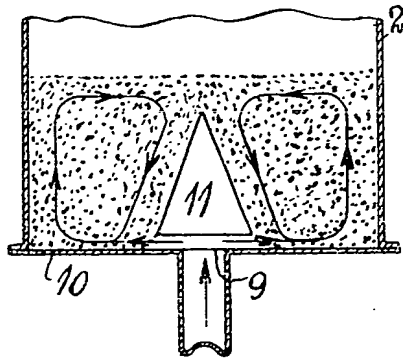


Fig. 6.

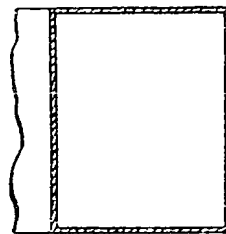
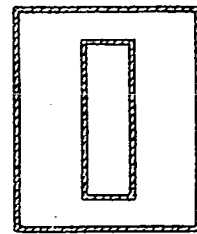


Fig. 8.



[This Drawing is a reproduction of the Original on a reduced scale.]

**THIS PAGE BLANK (USPTO)**